

[Research Title]: Electro-spun fine fibers of shape memory polymer used as an engineering part

Contractor (PI): Hirohisa Tamagawa

WORK Information:

Organization Name: Gifu University

Organization Address: 1-1, Yanagido, Gifu

Postal Code: 501-1193

Country: Japan

Work Phone Number: +81-58-293-2529

Work Fax Number: +81-58-230-1893

Work Email Address: tmgwhrhs@gifu-u.ac.jp

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14. ABSTRACT Electrospinning displays its high efficiency for fabricating a two-dimensional mat consisting of fine fibers. By the use of electrospinning, for example, a two-dimensional filter with superior functionality can be easily fabricated. However, individually fabricating fibers is a difficult task for the electrospinning technology, since electro-spun fibers are inevitably entangled and stick together during its formation process. Extraction of single fiber is quite difficult task, and establishing a single fiber extraction technique is a challenging theme in this field. This work introduces a quite simple technique of separately fabricating electro-spun short fibers. This technique simply employs a paper mesh, which is to be placed between the needle tip and counter electrode of electrospinning unit. Furthermore, usability of this technique was examined by try fabricating fine fibers consisting of different kinds of polymers.					
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ABSTRACT

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RESESRCH BACKGROUND

Figure 1 illustrates the setup of electrospinning apparatus. Highly charged polymer solution or melt loaded in a syringe is spewed out from the syringe needle and travels toward the counter electrode which serves as a fiber collectors because of the high electric field. Consequently, fine fibers are formed on the counter electrode. Those fibers formed are always laid on the counter electrode horizontally, and it is a benefit for fabricating a dense nonwoven fiber mat as illustrated in Figure 2. On the other hand, it becomes a drawback, when it comes to extracting fine fibers separately, since the fibers collected on the counter electrodes stick one another.

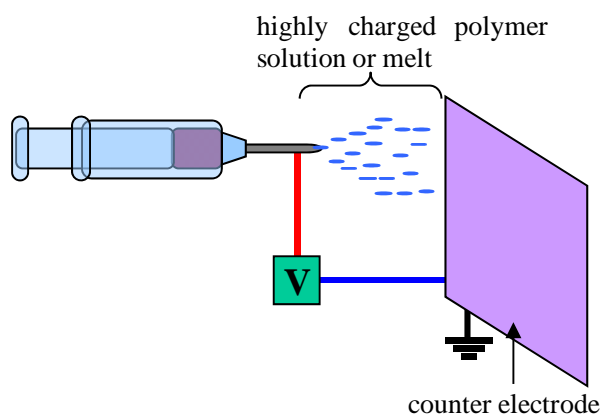


Figure 1 Setup of electrospinning.

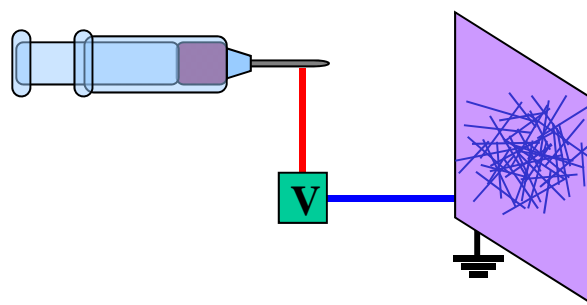


Figure 2 Fine fibers horizontally formed on the counter electrode forming a non-woven mat.

EXPERIMENTAL

It was observed in my electrospinning experiment that partially blocking the traveling path of highly charged polymer between the needle tip and counter electrode with a cardboard frame resulted in the formation of fibers between the cardboard frame and counter electrode vertically to the counter electrode. This phenomenon was employed for separately fabricating electro-spun short fibers

Polymer solution

Two kinds of polymer solutions were prepared to be electro-spun.

- **Polynorobornen** 2.5 g of polynorobornen – hereafter called PN for short – was dissolved into 50 g of THF in the water bath at 70 degree C. Once it was fully dissolved, it was cooled down to the room temperature.
- **Polyvinylacetate** 11.5g of polyvinyl acetate – hereafter called PVAc for short – was dissolved into 100g of

DMF in the water bath at 70 degree C. Once it was fully dissolved, it was cooled down to the room temperature.

Electrospinning setup

Conventional setup of electrospinning was already shown in Figure 1. For separately fabricating electro-spun fibers, a paper mesh (instead of a cardboard) was placed between the needle tip and counter electrode as illustrated in Figure 3.

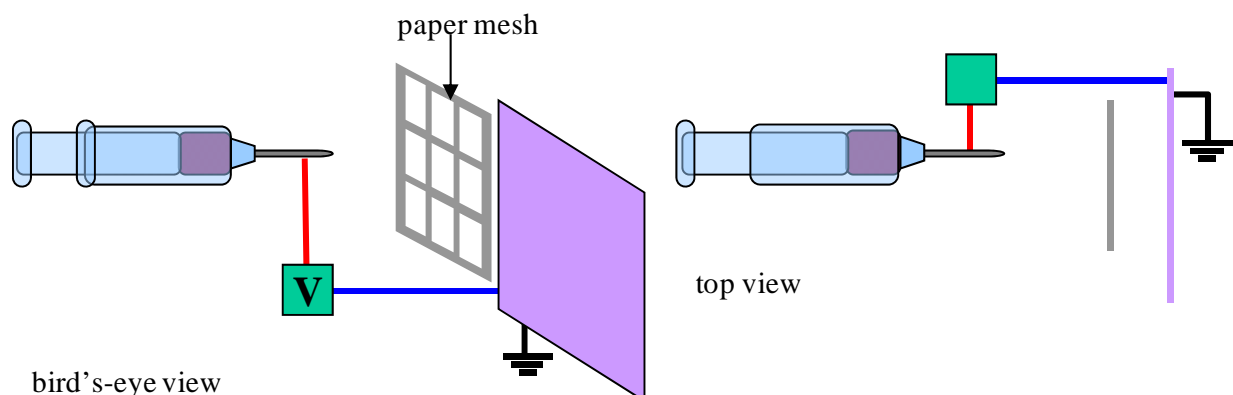
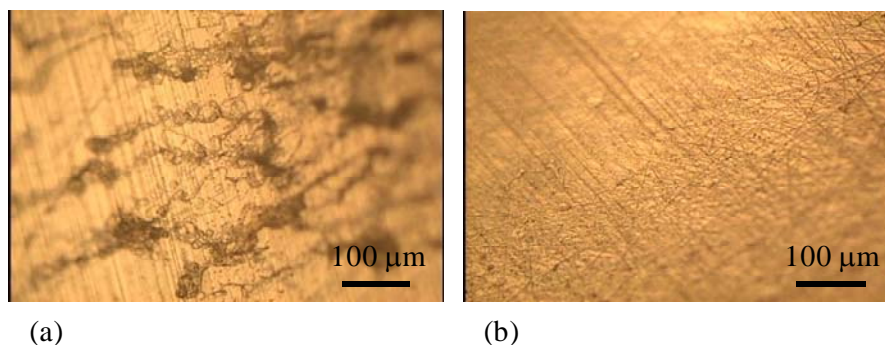


Figure 3 Setup for fabricating electro-spun fibers separately using a paper mesh.

RESULTS AND DISCUSSION

Fiber fabrication without a paper mesh

Using the setup without a paper mesh illustrated in Figure 1, the electrospinning of PN and PVAc was carried out, where the voltage was ~ 10 kV and the gap between the needle tip and counter electrode was ~ 10 cm. Figure 4 (a) and (b) respectively show the fibers of PN and PVAc formed on the counter electrode. Concerning PN fibers, they are highly entangled and seen as dark islands in Figure 4 (a). The PN fibers were formed horizontally to the counter electrode surface and stick together. These fibers could not be extracted separately. PVAc fibers were also formed on the counter electrode surface horizontally. Although those fibers are not seen so clearly in Figure 4 (b), relatively a large number of fine PVAc fibers are seen at the right bottom of Figure 4 (b). They stuck together and could not be extracted separately.



(a) (b)
Figure 4 Electro-spun (a) PN and (b) PVAc fibers formed horizontally on the counter electrode.

Separately fabricated short fibers

Using the setup with a paper mesh illustrated in Figure 3, electrospinning was carried out. The highly charged polymer solution in the syringe was spewed out toward counter electrode under the high voltage, where

the voltage was ~ 10 kV and the gap between the needle tip and counter electrode was ~ 10 cm. It passes through the paper mesh, resulted in the formation of fine fibers separately between the paper mesh and counter electrode. Figure 5 shows the PN fibers formed between the paper mesh and counter electrode. Compared with PN fibers shown in Figure 4 (a), the fiber diameter was quite large. Fiber diameter heavily depends on the condition of electrospinning such as voltage, gap between the needle tip and counter electrode, the ratio of PN and solvent and so on. Since it was difficult to precisely control the experimental condition, it was difficult to fabricate the same diameter fibers. However, it was repeatedly confirmed, this paper mesh method could be definitely used for fabricating the PN fibers separately.



Figure 5 PN fibers formed between the paper mesh and counter electrode.

Formation process of separately formed short fibers

Formation process of the separately formed short fibers is considered. Undoubtedly a quite simple process is brought to our mind as illustrated in Figure 6. Fiber ingredient is spewed out from the needle tip, and it travels toward paper mesh (Figure 6 (i)). It is trapped with the mesh (Figure 6 (ii)), and immediately it is stretched toward the counter electrode, resulting in the short fiber formation (Figure 6 (iii)). But actual process was a bit different. Using a high speed camera (ES Kodak EKT APRO HS Motion Analyzer Model 4540), the fiber formation process was analyzed.

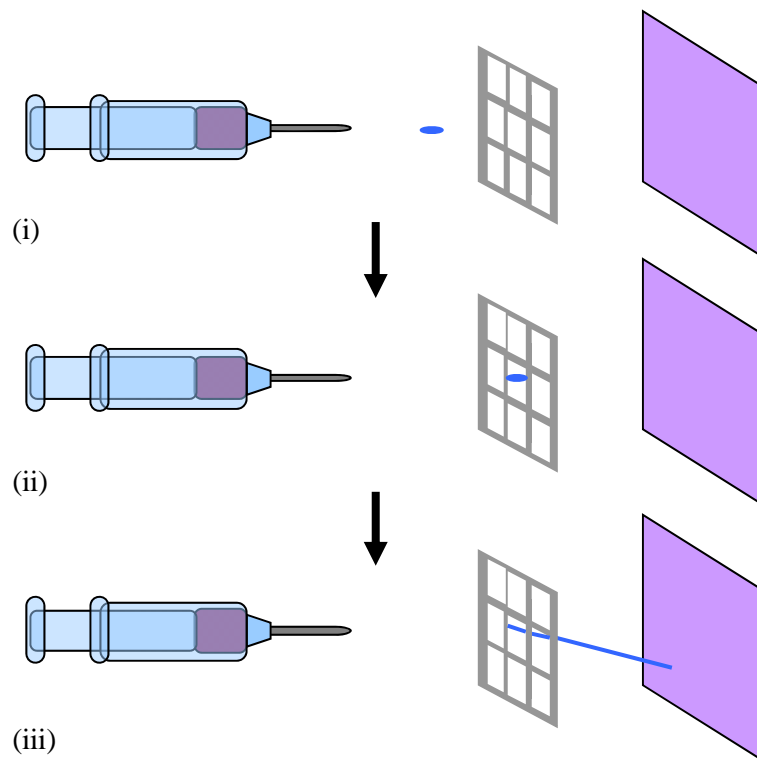


Figure 6 Formation process imagined for separately formed fibers between the paper mesh and counter electrode.

Figure 7 shows the time history of PN fiber formation process from 0 ms through 160 ms, where time $t = 0$ ms in Figure 7 (i) was arbitrarily defined. Once the voltage was applied between the syringe needle tip and counter electrode, the fiber ingredient was continuously supplied to the paper mesh as shown in the encircled area with solid line in Figure 7, where such a situation is roughly illustrated in the upper left of Figure 7 for better understanding. The polymer supply continued from beginning to end in this experiment. But fiber formation between the needle tip and counter electrode was not induced for a while even after the start of the polymer supply at $t = 0$ ms. Fiber formation suddenly started at $t = 40$ ms as shown in the encircled area with dotted line in Figure 7 (ii). The fiber continued to grow fat and it completed at $t = 160$ ms as shown in Figure 8 (v). During fiber growing process, the fiber initially kept on waving quite largely but it gradually subsided down to stillness. Figure 8 shows the waving motion of fiber. Figure 8 (a) and (b) were taken at $t = 56$ ms and 59 ms, respectively. The position of fiber in

Figure 8 (a) indicated by an arrow is obviously different from that in Figure 8 (b) because of rampant wavy motion of fiber.

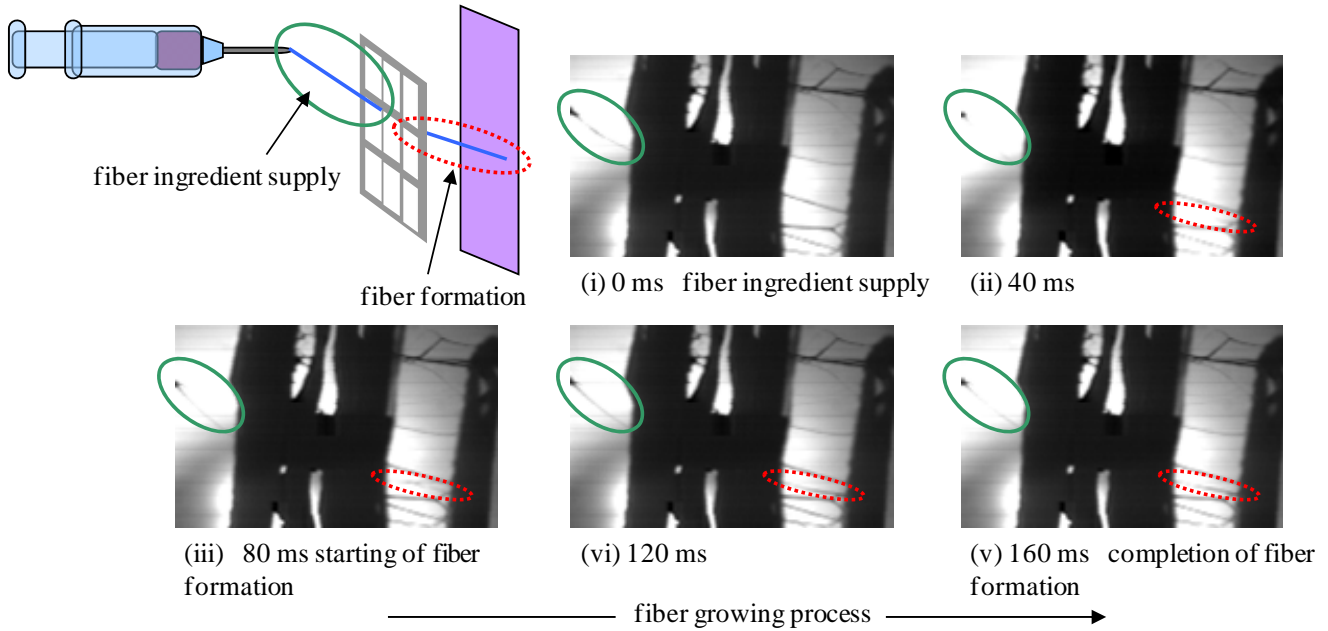


Figure 7 The time history of actual PN fiber formation process from 0 ms through 160 ms, where time $t = 0$ ms was defined arbitrarily.

The fiber formation was not continuous process. Firstly, the fiber ingredient accumulated on the paper mesh. Secondly, the fiber jet was suddenly spewed out from the paper mesh to the counter electrode, resulting in a fiber. Since there were a number of holes on the paper mesh, fiber formation was induced not only at one hole but at multiple holes. Even though the supply of fiber ingredient continued at single position of paper mesh as shown in Figure 8, the accumulated fiber ingredient flew to the multiple holes of paper mesh, eventually resulting in the fiber formation from those multiple holes. As clearly seen in Figure 8, a number of short fibers were formed between the multiple holes of paper mesh and counter electrode.

Use of this paper mesh technique did not result in so successful formation of PVAc fibers. It must be due to the less volatile property of DMF compared with THF not due to the ineffectiveness of this paper mesh technique. In fact, even a composite fiber consisting of PVAc dissolved in THF instead of DMF with Cu powder was well formed under the condition that the voltage was ~ 9 kV and the gap between the needle and counter electrode was ~ 4 cm as shown in Fig. 9.

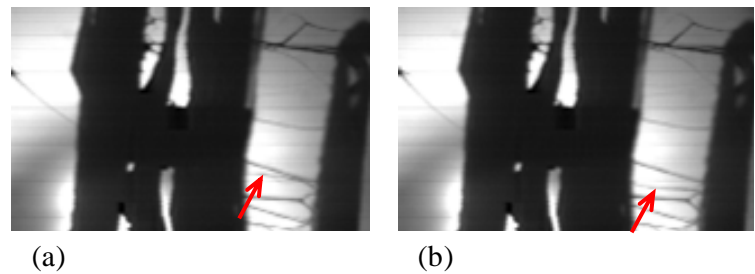


Figure 8 The waving motion of fiber at $t =$ (a) 56 and (b) 59 ms.

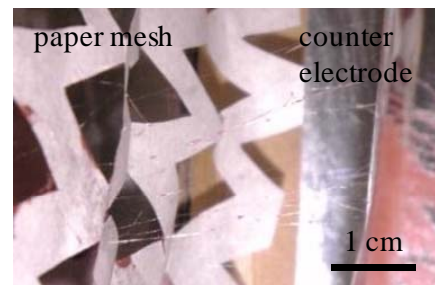


Figure 9 Electro-spun composite fibers consisting of PVAc and Cu powder formed between the paper mesh and counter electrode.

CONCLUSIONS

This work provided with a quite simple technique for fabricating the separately formed electro-spun fibers and elucidated the fiber formation process. Simply placing a paper mesh between the needle tip and collector resulted in the separately formed electro-spun polymer fibers, and it was observed that the fiber grows fat gradually its formation process.

This fiber fabrication technique was even applicable for the fabrication of polymer-metal composite fibers. However, it is important to use highly volatile solvent for the preparation of polymer ingredient to be electro-spun, otherwise the fibers are not effectively formed separately.

This technique must be useful for separately fabricating the broad range of different kinds of fibers.